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REAGENTS AND STANDARD SOLUTIONS FOR USE IN CHEMICAL ANALYSIS OF METALS, ORES AND MINERALS

PART I VOLUMETRIC SOLUTIONS

(First Reprint MARCH 1986)

UDC 543.24-4:622.34

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INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 2

July 1975

Indian Standard

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Indian Standard

REAGENTS AND STANDARD SOLUTIONS FOR USE IN CHEMICAL ANALYSIS OF METALS, ORES AND MINERALS

PART I VOLUMETRIC SOLUTIONS

0. FOREWORD

0.1 This Indian Standard (Part I) was adopted by the Indian Standards Institution on 27 June 1975, after the draft finalized by the Methods of Chemical Analysis Sectional Committee had been approved by the Structural and Metals Division Council.

0.2 A number of reagents and standard solutions are commonly used in volumetric, colorimetric and photometric determinations. This standard has been prepared with a view to ensuring uniformity in the description of the methods of preparation of these reagents and standard solutions in Indian Standards on methods of chemical analysis of metals, ores and minerals. While the preparation of primary and secondary standard solutions used in volumetric determination have been covered in this part, the methods for preparation of standard solutions used in colorimetric determination will be covered in Part II and those for commonly used reagents and indicator solutions are intended to be covered in Part III of this standard.

0.3 In the formulation of this standard due weightage has been given to international co-ordination among the standards and the practices prevailing in different countries in addition to relating it to the practices in the field in this country. This has been achieved by deriving assistance from '1973 Book of ASTM Methods for chemical analysis of metals, Part 32', issued by the American Society for Testing and Materials.

0.4 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

1. SCOPE

1.1 This standard (Part I) prescribes methods for preparation of standard solutions (primary and secondary) commonly used in volumetric determinations prescribed in Indian Standards on chemical analysis of metals, ores and minerals.

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*Rules for rounding off numerical values (*revised*).

2. QUALITY OF REAGENTS

2.1 Pure chemicals of analytical reagent quality shall be employed for the preparation of standard solutions, and distilled water (*see* IS : 1070-1960*) shall be used for this purpose.

SECTION I PRIMARY STANDARD SOLUTIONS

3. ARSENIOUS OXIDE (OR SODIUM ARSENITE) SOLUTION

3.1 Preparation of 0·1 N Solution — Dissolve 4·946 g of pure arsenious oxide (As_2O_3) previously dried at 105 to 110°C to constant mass in sodium hydroxide solution (20 percent). Dilute to about 200 ml and neutralize the solution with 1 N hydrochloric acid using litmus paper. Add 4 g of pure sodium bicarbonate. When the salt has completely dissolved, transfer the solution to a 1 000-ml volumetric flask, dilute with water to the mark and mix.

4. EDTA SOLUTION

4.1 Preparation of 0·05 M Solution — Dissolve 14·62 g of ethylenediamine-tetraacetic acid in 50 ml of 1 N sodium hydroxide solution and dilute to 1 000 ml. Store in a polythene bottle.

4.2 Preparation of 0·05 M Solution — Dissolve about 18·64 g of the disodium salt of EDTA [$(\text{OOC.CH}_2)_2\text{NH.CH}_2\text{CH}_2\text{.NH}(\text{CH}_2\text{COO})_2\text{Na}_2\text{.2H}_2\text{O}$] in water and transfer to a 1000-ml volumetric flask, dilute to the mark and mix.

5. POTASSIUM BROMATE SOLUTION

5.1 Preparation of 0·1 N Solution — Dissolve 2·784 g of potassium bromate ($KBrO_3$), previously dried at about 150°C to constant mass in water and transfer to a 1 000-ml volumetric flask. Dilute to the mark and mix.

6. POTASSIUM DICHROMATE SOLUTION

6.1 Preparation of 0·1 N Solution — Dissolve 4·904 g of potassium dichromate ($K_2Cr_2O_7$), previously dried at about 150°C to constant mass in water and transfer to a 1 000-ml volumetric flask. Dilute to the mark and mix.

7. POTASSIUM HYDROGEN PHTHALATE SOLUTION

7.1 Preparation of 0·1 N Solution — Dissolve 20·42 g of potassium hydrogen phthalate ($HKC_8H_4O_4$) previously dried at 120°C to constant mass in freshly boiled and cooled water and transfer the solution to a 1 000-ml volumetric flask. Dilute with water to the mark and mix.

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*Specification for water, distilled quality (*revised*).

8. POTASSIUM IODATE SOLUTION

8.1 Preparation of 0·1 N Solution for Antimony Titration in Strong Acid Medium (3M to 9M) — Dissolve 5·351 g of the finely powdered potassium iodate (KIO_3), previously dried at 120°C, in about 400 ml of water and dilute with water to 1 000 ml.

8.2 Preparation of 0·1 N Solution (for Iodometric Titration) — Dissolve 3·567 g of potassium iodate, previously dried at 160 to 180°C to constant mass, in 200 ml of water. Add 1 g of sodium hydroxide and 10 g of potassium iodide. Transfer the solution to a 1 000-ml volumetric flask, dilute with water to the mark and mix.

9. SODIUM CARBONATE SOLUTION

9.1 Preparation of 0·1 N Solution — Dissolve 5·300 g of the previously dried material (by heating at about 260°C to constant mass) in water; transfer the solution to a 1 000-ml volumetric flask. Dilute with water to the mark and mix.

10. SODIUM CHLORIDE SOLUTION

10.1 Preparation of 0·1 N Solution — Dissolve 5·845 g of previously dried sodium chloride (NaCl) in water and transfer to a 1 000-ml volumetric flask, dilute to the mark and mix.

11. ZINC SOLUTION

11.1 Preparation of 0·1 M Solution — Dissolve 6·538 g of electrolytic zinc granules (99·95 percent purity) in dilute hydrochloric acid (1 : 1) and transfer the solution to a 1 000-ml volumetric flask, dilute with water to the mark and mix.

SECTION 2 SECONDARY STANDARD SOLUTIONS

12. FERROUS AMMONIUM SULPHATE SOLUTION

12.1 Preparation of 0·1 N Solution — Transfer about 40 g of ferrous ammonium sulphate crystals [($FeSO_4 . (NH_4)_2 SO_4 . 6H_2O$)] to a 1 000-ml volumetric flask. Add 500 ml of dilute sulphuric acid solution (5 : 95) shake well until the solid has dissolved and make up to the mark, on cooling, with dilute sulphuric acid (5 : 95). Standardize the solution against any suitable oxidizing agent, such as potassium dichromate, or potassium permanganate solution.

13. HYDROCHLORIC ACID

13.1 Preparation of 0·1 N Solution — Transfer by means of a graduated cylinder or a burette, 9 ml of concentrated hydrochloric acid ($d = 1·16$) into a 1 000-ml volumetric flask containing about 500 ml of water. Cool, dilute with water to the mark and mix. Standardize the solution against standard sodium carbonate solution.

14. IODINE SOLUTION

14.1 Preparation of 0·1 N Solution — Transfer 12·7 g of resumblimed iodine to a 250-ml beaker, containing 25 g of iodate-free potassium iodide and 25 ml of water. Stir until all the iodine is dissolved and dilute with water to 1 000 ml. Preserve in an amber-coloured glass-stoppered bottle in a cool place. Standardize against a standard solution of sodium arsenite, sodium thiosulphate, etc.

15. NITRIC ACID

15.1 Preparation of 0·1 N Solution — Transfer by means of a graduated cylinder or a burette 7 ml of concentrated nitric acid ($d = 1{\cdot}42$) into a 1 000-ml volumetric flask containing about 500 ml of water. Cool, dilute with water to the mark and mix. Standardize the solution against standard sodium carbonate solution.

16. POTASSIUM FERROCYANIDE SOLUTION

16.1 Preparation of 0·05 M Solution — Dissolve 21·12 g of potassium ferrocyanide [$K_4Fe(CN)_6{\cdot}3H_2O$] and 0·5 g of potassium ferricyanide in freshly boiled water and dilute to 1 000 ml. Standardize against standard zinc solution.

17. POTASSIUM PERMANGANATE SOLUTION

17.1 Preparation of 0·1 N Solution — Dissolve 3·2 g of potassium permanganate in 1 litre of water. Let stand in the dark overnight. Filter, without washing, through glass wool. Avoid contact with rubber or other organic material. Store in a dark-coloured glass-stoppered bottle. Standardize against standard sodium oxalate solution.

18. SILVER NITRATE SOLUTION

18.1 Preparation of 0·1 N Solution — Dissolve 17 g of silver nitrate ($AgNO_3$) in water and dilute to 1 000 ml. Store in an amber-coloured glass-stoppered bottle. Standardize against standard pure sodium chloride.

19. SODIUM HYDROXIDE SOLUTION

19.1 Preparation of 0·1 N Solution — Dissolve 50 g of sodium hydroxide (NaOH) in 50 ml of water in polythene beaker and transfer to a large test tube; avoid wetting the top. Stopper tightly and let stand in a vertical position until the supernatant liquid is clear. Carefully withdraw and transfer 5·0 ml of the clear solution to a 1-litre volumetric flask containing about 750 ml of freshly boiled and cooled water, dilute to volume with more of the freshly prepared water, and mix. Standardize before use against standard potassium hydrogen phthalate solution.

20. SODIUM THIOSULPHATE SOLUTION

20.1 Preparation of 0·1 N Solution — Dissolve 24·8 g of sodium thiosulphate ($Na_2S_2O_3$, $5H_2O$) in water containing 0·5 g of sodium carbonate or 2 ml of chloroform and dilute to 1 litre. Keep it overnight. Standardize against standard potassium iodate solution.

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